Location: QA Director's Office
SOP Files
Analyst's work bench Conventional Laboratory #103-7

1.0 SCOPE

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.2 The method is suitable for all concentration ranges; however, appropriate aliquots should be used to avoid a titration volume greater than 50-mL.

2.0 SUMMARY OF THE METHOD

2.1 This SOP is a procedure for evaluating Alkalinity in liquid samples.

3.0 INTERFERENCES

3.1 N/A

4.0 APPARATUS AND MATERIALS

4.1 Digital titrator

4.2 Stir plate

4.3 Clear plastic cups

5.0 REAGENTS

5.1 Deionized (DI) water

5.2 0.1600 sulfuric acid titration cartridge

5.3 Bromcresol Green-Methyl Red indicator pillow packets

5.4 25,000-mg/L alkalinity standard

6.0 SAMPLE HANDLING AND PRESERVATION

6.1 Alkalinity is best analyzed as soon as possible from the time the sample is collected. Maximum holding time for **water samples from the time of sampling is 14 days** refrigerated at $4^{o}C \pm 2^{o}C$

6.2 Samples should be provided in unpreserved, clear plastic bottles.

7.0 PROCEDURE [1], [2]

7.1 COLOR DEVELOPMENT AND MEASUREMENT (TOTAL)

7.1.1 Pour 100-mL of DI water in a clear plastic cup to make a Blank sample. Place a magnetic stirring bar in the cup and place the cup on a stir plate. Turn on stir place so that the sample is stirred well, but its color is still clearly visible.

7.1.2 Pour one powder pillow in the cup.

7.1.3 Using a digital titrator set up with a 0.1600 sulfuric acid titration cartridge, place its tip in the sample so that the acid may be released into the sample. Slowly turn the endpiece clockwise until the water just turns from its original green color to pink/purple.

7.1.4 The number of digits used to do this is the value for the Blank. This value will be subtracted from the digit values of all other samples.

7.1.5 Rinse off magnetic stir bar.

7.1.6 Place 5-mL of a sample in another cup and add 95-mL DI water to it. Repeat the same procedure to find the number of digits needed to titrate the sample. If this value is exceedingly small or large, the dilution of the sample may be adjusted.

7.2 CARBONATE AND BICARBONATE MEASUREMENT

7.2.1 Place 5-mL of sample in cup, add the contents of a Phenolpthalein Indicator Powder pillow and swirl to mix.

7.2.1.1 If there is no color change, dilute and titrate exactly like a Total Alkalinity sample.

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[1] *Standard Methods*, twentieth edition, Method 2320B, Alkalinity.

[2] Hach Digital Titrator Model 16900-01 Manual. Alkalinity.

7.2.1.2 If there is a color change to pink, dilute and titrate to a colorless endpoint. Record the number of digits required as the Phenolpthalein Alkalinity. (Do not reset digit counter!) Continue titration same as Total Alkalinity, adding the contents Bromcresol Green-Methyl Red indicator pillow packet to the sample. Record the number of digits required.

8.0 CALCULATIONS

8.1 Total Alkalinity, $\frac{mg}{L}$ $CaCO_3$ = $\frac{D*100mL*M}{S}$

Where: D = number of digits used in titration of sample
M = multiplier number specified by concentration of cartridge (in this case, 0.1)
S = mL of sample used

8.2 Bicarbonate Alkalinity, $\frac{mg}{L}$ = Total Alkalinity ($\frac{mg}{L}$) – (2 * Phenolpthalein Alkalinity)

8.3 Carbonate Alkalinity, $\frac{mg}{L}$ = 2 * Phenolpthalein Alkalinity

9.0 QUALITY CONTROL

9.1 See Table 1

9.2 Samples are analyzed in batches of thirty or less per QC set. The QC samples that are analyzed per batch are:

- Standard Check
- MS
- LCS
- DUP
- MSD (optional)
- Method Blank

10.0 DOCUMENTATION

10.1 Bench book for digestion and analysis

10.2 Work sheets

11.0 METHOD PERFORMANCE

1.1 Precision and accuracy studies are performed on as needed basis. (Ex. new instrument, etc.)

1.2 Method Detection limit studies are performed annually.

Table 1. Quality Control Requirements (Sample Set = 20 samples)

QC Analysis	Required/ Frequency	Limits	Corrective Action	Corrective Action after Reanalyzing
Method (preparation) Blank	Yes One each set	<MDL or 1/10 Regulatory limit	Remove contamination and rerun	
Laboratory Control Sample (LCS) Soluble or insoluble	Yes One every 20 samples	85%-115%	Rerun	
Matrix Duplicate	Yes One each set	RPD<20%	Rerun entire set	
Matrix Spike	Yes One each set	85%-115%	Analyze by Method of Standard Additions	
Matrix Spike Duplicate	Yes One every 20 samples			
Dilution & Rerun	No except if result indicates suppressive interference	Does interference persist?	Yes. Rerun with Method of Standard Additions	

12.0 APPROVAL & ISSUE:

12.1 The following personnel have read, accepted and approved this standard operating practice.

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Analyst Date

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Andy Ball, QA Officer Date

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Maya V. Murshak, QA Director Date